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A RESEARCH INVESTIGATION OF POSSIBILITIES FOR OBTAINING HOT-HARD ELECTRODEPOSITED CHROMIUM OR CHROMIUM-BASE ALLOYS FOR CANNON

to

WATERTOWN ARESENAL

February 9, 1952

FINAL TECHNICAL REPORT

on

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J. Edwin Bride, George M. Scanlon, Cloyd A. Snavely, and Charles L. Faust

Contract No. DA-33-019-ORD-9
W.A.L. File No. 691.1/25-42
O.O. Project No. TR3-3003B

BATTELLE MEMORIAL INSTITUTE
505 King Avenue
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FINAL TECHNICAL REPORT

Contractor: Battelle Memorial Institute

Agency: Office, Chief of Ordnance, ORDTR-Cannon

Ordnance District: Cleveland, Ohio

Contract Number: DA-33-019-ORD-9 W.A.L. File No. 691.1/25-42

O.O. Project Number: TR3-3003B

Priority: War Department 2B

Title of Project: "A Research Investigation of Possibilities for Obtaining Hot-Hard Electrodeposited Chromium or Chromium-Base Alloys for Cannon".

Authors: J. Edwin Bride, George M. Scanlon, Cloyd A. Snavely, and Charles L. Faust.

Object: To investigate possibilities for an erosion-resistant chromium or chromium-alloy electroplate for lining gun tubes.

Summary: Experiments were continued on the application of 94 per cent chromium, 6 per cent iron alloy plate to the bore surfaces of cannon. After moving-anode tests were unsuccessful, a satisfactory technique was developed using full-length anodes in the bore of 4-foot-long sections of 40-mm gun tubes. Plates were then produced with good appearance, good plating efficiency, and adequate dimensional control, but the adhesion to the base metal was not good enough for gun-tube service. Several erosion-gage weapon inserts were plated and test fired. Each test resulted in failure by separation of the plate from the bore surface. Some improvement in adhesion was achieved by a thin chromium plate between the steel base and the chromium-iron alloy plate. A bonding heat treatment also effected some improvement. However, erosion-gage weapon-firing tests showed that adhesion was still not sufficient to withstand the conditions encountered in gun-tube service.

Conclusions and Recommendations

While the results of the erosion-gage weapon-firing tests were negative and show that further improvements are necessary, the outlook for final success continues to be encouraging. Considerable progress has been made in development of techniques for plating the chromium-iron alloy in

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SCOPE OF WORK

The work reported herein falls under the following general headings:

a. An investigation of the possibilities for electrodepositing new alloys of chromium which are hot hard and crack free.

b. A continuation, as necessary, of recent work relating to the development of electrodeposited chromium-iron alloys. This work includes "beaker-scale" application to gun-tube sections, though not to whole gun tubes.

c. Recommendation of methods for application to cannon of any significant new developments under "a" and "b", if such developments are completed to a suitable stage.

The supplemental agreements to the original contract provided for an accelerated effort to continue the work outlined above and to establish instructions and specifications for applying any significant new developments to cannon.

plating took place along the entire length of the tube at one time. However, new techniques of solution control were necessary and the tube-plating work was recessed from time to time in order to do beaker-scale work needed for guidance in modifying the tube-plating operations.

Much of the tube-plating work was done with 4-foot lengths of simulated 40-mm smooth-bore tube. Several 4-foot rifled 40-mm tube sections were furnished by Watertown Arsenal for plating tests. A number of short (3- to 4-inch) sections of rifled tube were used for smaller scale tests. In addition, erosion-gage weapon inserts were plated at intervals when it appeared that success in firing tests was possible. In each case, the plates failed early in the tests by separation from the bore surface.

The problem of adhesion of the alloy plate to the steel tube surfaces was attacked then and as yet has not been solved entirely. A thin plate of conventional chromium on the steel, followed by the desired thickness of alloy plate, gave better performance in bend tests and chisel-gouging tests used for preliminary evaluation. A bonding heat treatment at 750 F for one hour effected additional improvement. However, firing tests demonstrated that this technique effected little or no improvement over plating the alloy directly on steel.

Three Interim Technical Reports* describe in detail the tests mentioned in the foregoing discussion, except for those performed in the last six months of work, which are reported fully herein. In the following sections, the critical experiments are described in sufficient detail to clarify their influence on the work that followed, and the final six months' work is detailed completely for purposes of record and to delineate the present position of this research.

EXPERIMENTAL

Moving-Anode Tests

Method of Attack

A pilot-scale plating unit was constructed for plating the bore surfaces of tubes up to 18 inches long. The unit was used mainly for moving-anode tests, either with or without diaphragms. Later, portions of the unit were used in plating 4-foot lengths of tube.

*Ordnance Contract DA-33-019-ORD-9, "A Research Investigation of Possibilities for Obtaining Hot-Hard Electrodeposited Chromium or Chromium-Base Alloys for Cannon", Interim Technical Reports dated September 1, 1950, September 15, 1951, and January 15, 1952.

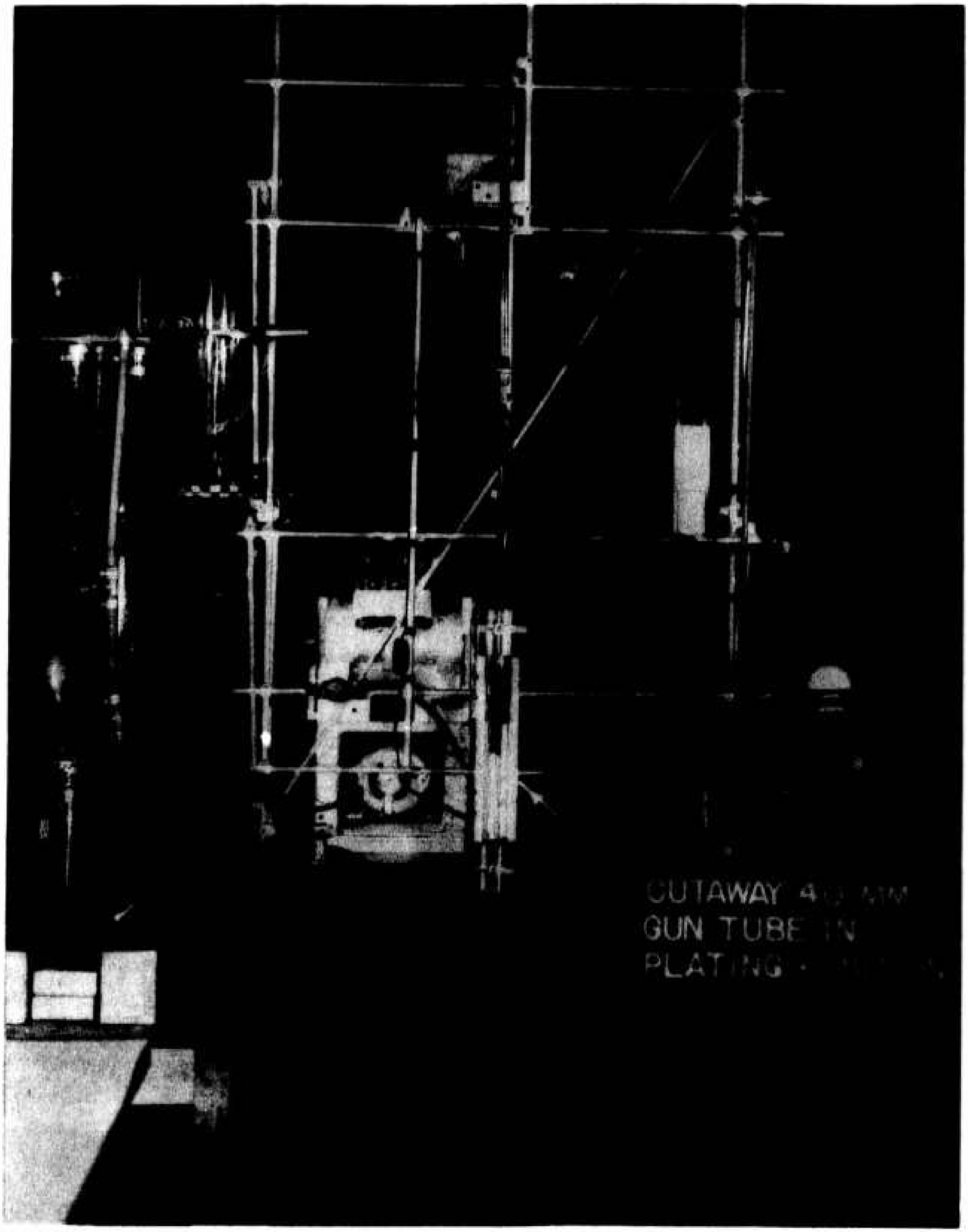

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FIGURE 1. PHOTOGRAPH OF PILOT UNIT FOR PLATING GUN-TUBE SECTIONS WITH MOVING ANODE

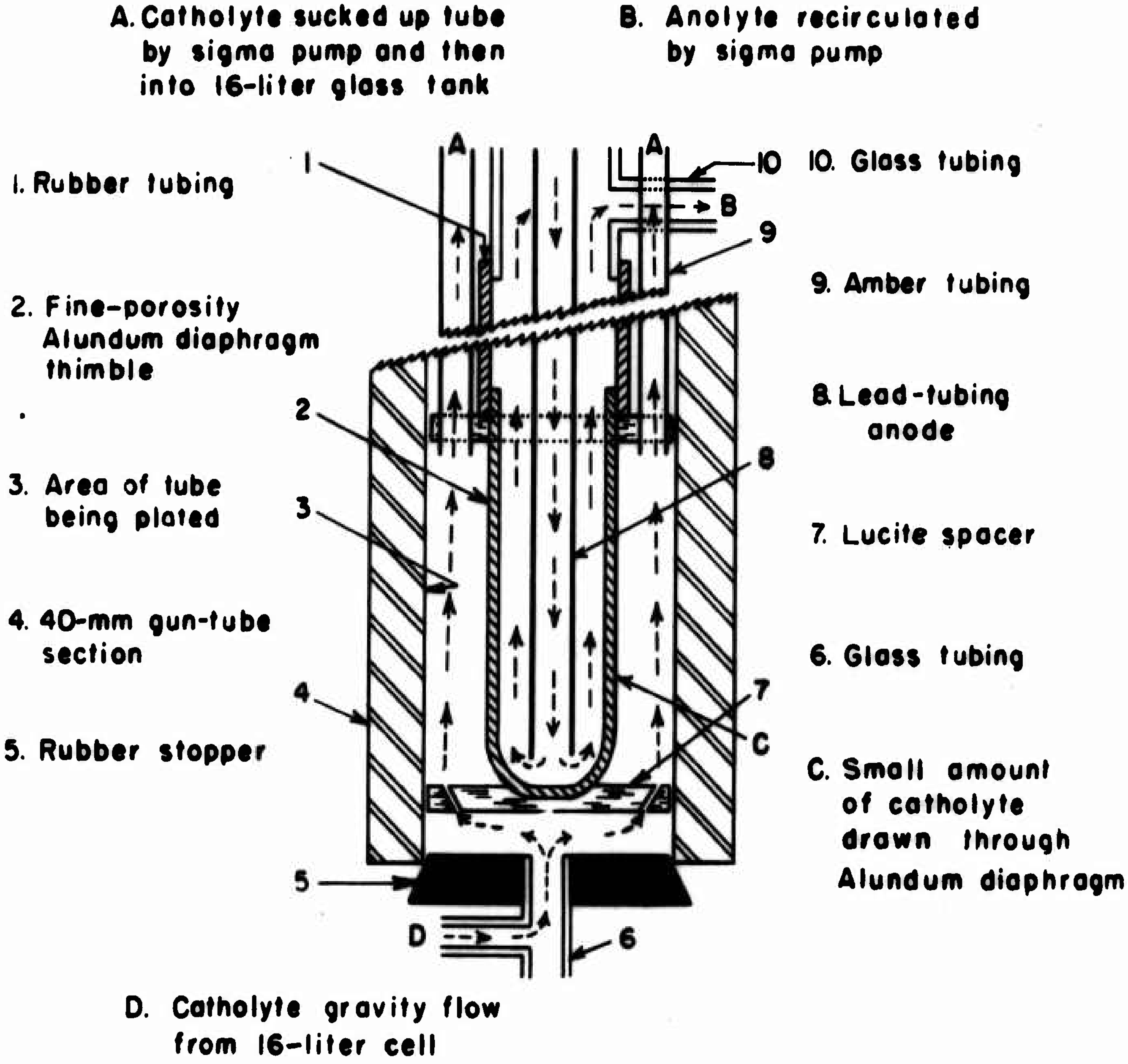


FIGURE 3. INSOLUBLE ANODE ASSEMBLY WITH DIAPHRAGM

0-18404

Standard Bath Formulation (As Modified).

Ammonium Hydroxide (28%) NH_4OH	- 60 ml/l
Chromium Ammonium Sulfate $Cr_2(SO_4)_3(NH_4)_2 \cdot SO_4 \cdot 24H_2O$	-700 g/l
Ferrous Ammonium Sulfate $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$	- 13.5 g/l
Magnesium Sulfate $MgSO_4 \cdot 7H_2O$	- 20 g/l
Ammonium Sulfate $(NH_4)_2SO_4$	- 50.0 g/l
Sodium Sulfite (Stock Solution Containing 0.005 g/ml) Na_2SO_3	- 50 ml/l
Duponol M.E.*	

The optimum operating conditions were as follows:

Electrolyte Flow (Through 1.5-Inch-ID Tubes)	1 to 2 liters/min
Cathode Current Density	375 to 400 amp/sq ft
pH (at 145 F)	1.4 to 1.7
Bath Temperature	140 to 150 F

The work progressed through various tests relating to the amount of heat transfer to be considered when plating at high current densities, the best electrolyte-flow rate, the anode-travel rate, and the pH control. As indicated by the results, the various anode designs previously described were devised and tested. The best results were obtained after discovering that the porous diaphragm separating the anode and cathode could be eliminated. Any oxidation products formed at the anode were reduced chemically by the addition of hydrogen peroxide to the plating reservoir tank. Sodium sulfite was shown to be an unsatisfactory reducing agent when it was necessary to use it in large amounts, rather than as a minor addition agent, as given in the Standard Bath Formulation. Elemental sulfur appeared in the bath as a serious contaminant.

*Surface-active agent manufactured by E. I. du Pont de Nemours and Company, Wilmington, Delaware.

Two rifled erosion-gage weapon inserts were used in preliminary tests to establish plating conditions. Full-length, lead-tin-coated copper anodes were used, centered by Lucite fittings at each end of the insert. Electrolyte flowed through the tube by gravity from a reservoir tank. Hydrogen peroxide was added to the reservoir as necessary to reduce hexavalent chromium formed at the anode during plating. Gas from the reduction reaction tended to "vapor lock" the tube carrying the electrolyte to the insert. An attempt to carry out the reduction of hexavalent chromium in a packed column did not eliminate the gas release in the tube to the insert.

A more successful plating method was devised then wherein the inserts with anodes affixed simply were suspended in a plating tank. The gas from the anode and cathode provided a "gas lift" to pump fresh solution through the tube continuously. A small portion of the solution was circulated through the reducing column and treated with hydrogen peroxide.

Inserts 59X and 60X were plated by this technique. Several trials were unsatisfactory, so the inserts were stripped and replated. Firing tests on the best plates were made at Watertown Arsenal. The adhesion of the plate was very poor.

Plating Tests With 4-Foot Lengths of Simulated 40-Mm, Smooth-Bore Gun Tube

Numerous plating tests were made using 4-foot lengths of simulated 40-mm smooth-bore gun tube. Full-length copper-rod anodes were plated with 90 per cent lead, 10 per cent tin alloy using a fluoborate solution. The chromium-iron alloy-plating electrolyte was pumped from a 40- to 50-liter storage bath, through the simulated 40-mm gun-tube sections, and overflowed back into the storage tank.

Wide ranges of electrolyte-flow rate, current density, and bath formulation were studied in the attempt to reach a set of conditions which would give a uniform and adherent plate over the entire bore surface. That goal was not reached in this series of tests. The best sample produced had a slightly gas-streaked area at the top. It had a plate of 0.003-inch thickness at the bottom and of 0.002-inch thickness about 15 inches from the top. A saw cut through the sample was thought at the time to demonstrate good adherence. However, later results showed that a saw cut is not a severe enough adherence test for electroplate for gun tubes.

The net results of this work were to show that plating with a full-length anode had considerably more promise than plating with a moving anode; that adhesion of the plate must be improved; and that more should be learned about bath control. The results were encouraging of final success, though not particularly successful in themselves.

TABLE 1. EVALUATION OF VARIOUS TREATMENTS AFFECTING THE ADHESION OF THE CHROMIUM-IRON ALLOY ELECTROPLATE ON STEEL

Test Number	Cathode Material	Variable to be Studied	Cathode Current Density, amp/sq ft	pH, Cr-Fe Bath	Plating Time, min	Pretreatment: Electro-polish	Pretreatment: Copper Strike Plate	Pretreatment: Brass Strike Plate	Results of Adhesion Tests (Bending)*: As Plated	After Annealing Treatment: 500 F, 15 hr	After Annealing Treatment: 750 F, 12 hr	After Annealing Treatment: 1000 F, 15 hr
Bath Composition: Bath No. 5977-39 used in the following tests. Refer to Table 2 for detailed composition formulation.												
5977-61A	Steel tube, 1.6" ID x 8"	Brass undercoat	350	1.55	30	--	--	1/2 of length	3	6	9	9
-61B	Ditto	Copper undercoat	"	--	"	--	x	--	4	8	8	8
-62A	"	Electro-polish	"	1.55	"	x	--	--	0	2	1	0
-62B	"	E.P. and Cu strike	"	--	"	x	x	--	2	6	7	1
-63A	"	Ditto	"	1.5	"	x	x	--	3	3	5	6
-63B	"	E.P. and brass strike	"	--	"	x	--	x	7	5	10	10
-68A	"	Anodic vs. cathodic cleaning	"	--	"	--	--	--	10	9	10	10
-68B	"	Lower c.c.d.	210	--	50	--	--	--	9	7	10	10
-70A	"	Copper strike plate	"	--	"	--	x	--	4	4	7	10
-71A	"	Brass strike plate	"	1.3	60	--	--	x	8	--	--	--
-72A	"	Raise pH	"	2.0	"	--	--	--	2	--	--	--
-72B	"	Raise pH and c.c.d.	350	2.15	40	--	--	--	--	--	--	--
-72C	"	Lower pH	350	1.6	"	--	--	--	--	--	--	--

*Numerical evaluations go from 0 = poor to 10 = very good.

TABLE 2. PLATING OF EROSION-GAGE WEAPON INSERTS 61X, 62X, and 63X

		Nickel-Strike Plating Conditions				Immersion	Chromium-Iron Alloy Plating				
Test Number	Insert Number	Cathode Current Density, amp/sq ft	Bath Temperature, F	Plating Time, min	Volts	Time in Cr-Fe Bath, No Current, min	Bath Temperature, F	pH	Cathode Current Density, amp/sq ft	Plating Time, min	Remarks
6208-62A	63X	100	106	5	3	1 min	140	1.15	400	30	Deposit appeared satisfactory
-62B	62X	"	"	"	"	Ditto	"	"	"	"	Plate not satisfactory - contains several blisters
-62C	61X	"	"	"	"	"	"	"	"	"	Deposit satisfactory; one very small raised spot
-63A	62X	"	"	"	"	"	"	"	"	"	Deposit from Test 6208-62B stripped; generator exciter trouble caused interruption in Cr-Fe plating; Deposit 63A slightly blistered
-65A	"	"	"	"	"	"	"	"	"	"	Deposit from Test 6208-63A stripped; Insert 62X given a hydrogen relief treatment of 400 F for 2 hours; deposit of this test (65A) also blistered
-65B	"	"	"	"	"	"	"	"	"	"	Deposit from Test 65A stripped; new chromium-iron bath made up; Deposit 65B appeared very good - no blisters or rough areas

in the alloy bath at 144 amp/sq ft after a 10-minute chromium strike and the deposits were adherent before and after heat treating.

The best adhesion of the chromium-iron alloy was obtained by employing a 2- to 10-minute chromium strike. This test series did not show that the bonding heat treatment is necessary for good adhesion, as was shown in later tests.

The cathodes used in the next series of adhesion tests were prepared by sawing 1/4-inch slices from the centerless-ground bar (17" long x 1-1/8" diam) of SAE 4140 steel, heat treated to a hardness of 27 R_c as furnished by Watertown Arsenal. One flat side of each 1/4-inch slice was given a 320-grit finish. A cathode connection was made by wrapping a double turn of thin iron wire around the cylindrical surface of the specimen. Plating tests were carried out in 4- to 5-1/2-liter chromium-iron alloy-plating baths formulated from basic chromic sulfate salts. Data for each test are given in Table 4.

In Tests Nos. 6526-8B, -9A, and -10C, the 4140 steel was given a reverse etch treatment in a 115 F caustic solution containing 120 g/l NaOH for 5 minutes at 40 amp/sq ft. A similar pretreatment has been used in preparing cast iron for chromium plating. The 4140 steel sample then was chromium-iron plated. The chisel-gouging test* indicated that adhesion was still not satisfactory. Then the plated sample was subjected to a 600 F treatment in hydrogen for 2 hours. A similar chisel-gouging test indicated improved adhesion. However, Test Samples 6526-9A and -10C, prepared and heat treated in a similar manner, did not confirm the improved adhesion of Test 6526-8B.

In plating Tests 6526-8A to -13A, the 4140 specimens were given a reverse etch in a conventional chromic acid plating bath and then plated for 2 to 10 minutes. This pretreatment eliminated the possibility of a smut film's forming at the chromium strike-4140 steel interface. The chromium-plated specimen was immersed in the chromium-iron plating bath with the current off. This procedure would be advantageous for plating gun tubes, as sufficient time could be allowed for the part to come to bath temperature.

The usual test for adherence has been to try to chip or flake the chromium-strike plate and the chromium-iron deposit from the 4140 basis metal by chisel gouging. The results of Test 6526-20A are typical. They indicate that adhesion was not satisfactory. However, when the plated 4140 specimen was subjected to a bonding heat treatment of 750 F for 2 to 3 hours in an air atmosphere, the chisel-gouging technique failed to flake the plates from the basis metal.

*The chisel-gouging test consists of cutting a chip out of the plate surface with a round-nosed cold chisel. The edges of the "gouge" so made are examined for evidences of flaking or peeling.

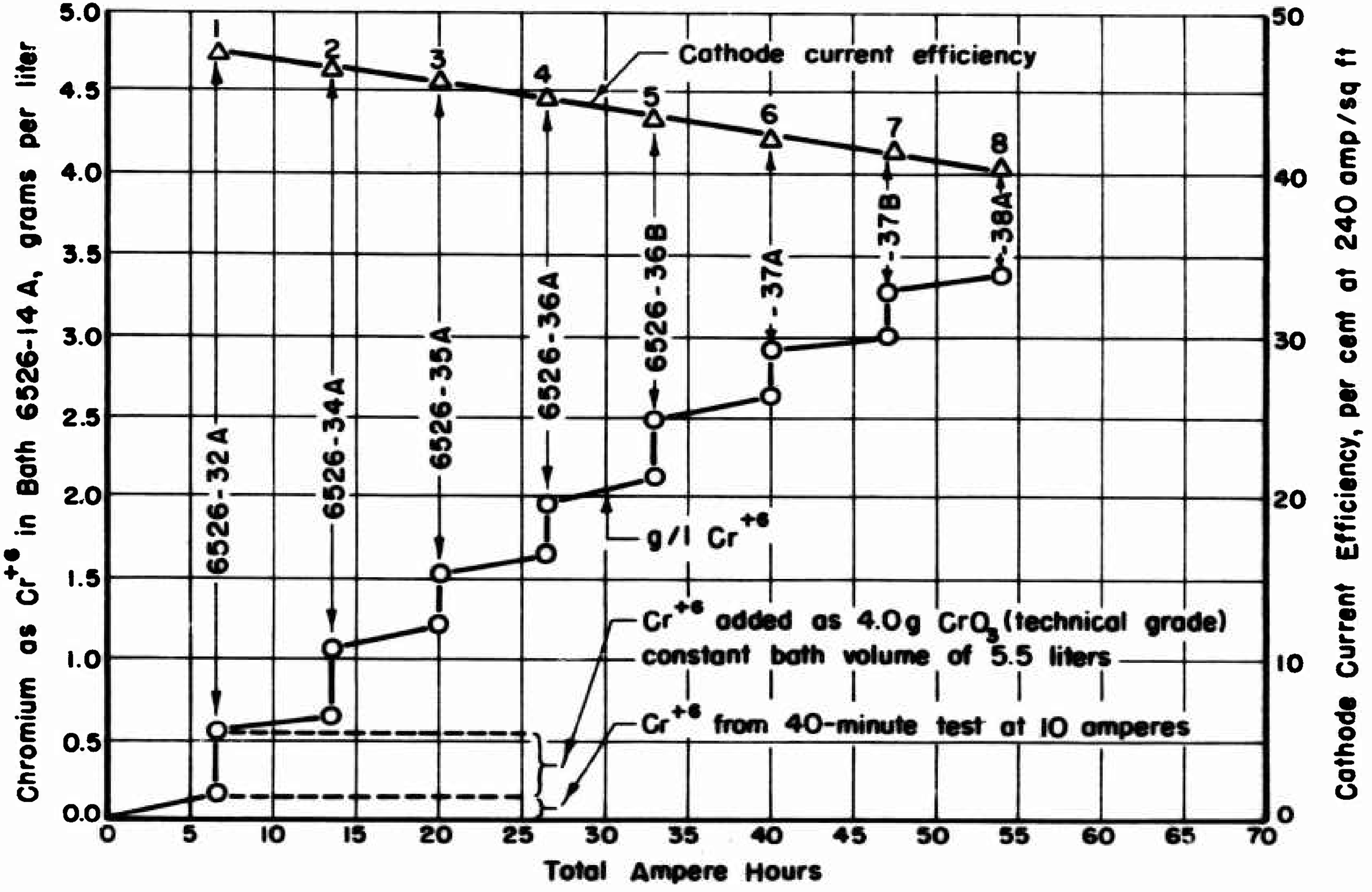


FIGURE 5. EFFECT OF HEXAVALENT CHROMIUM ON THE CATHODE CURRENT EFFICIENCY OF A CHROMIUM-IRON PLATING BATH

A-2688

Plating of 3-Inch-Long Rifled 40-Mm Gun-Tube Sections in the 100-Gallon Alloy Bath

Two 3-inch-long rifled sections of 40-mm gun tube and one 8-inch-long section were chromium-iron plated in the 100-gallon plating bath in order to evaluate the performance of this bath for further tests. The procedures for pretreatment, chromium strike, chromium-iron plating, and bonding heat treatment are outlined in Appendix III.

After the bonding heat treatment, the plated sections were checked for adhesion by gouging with a chisel. The adherence was considered satisfactory.

A small plated section was removed prior to the bonding heat treatment and mounted for metallographic study. A photomicrograph of the chromium-iron deposit in the "as polished" and "before heat treat" conditions is shown in Figure 6. The etched structure is shown in Figure 7. Figures 8 and 9 show that the bonding heat treatment has very little, if any, noticeable effect on the plate structure. The chromium-strike deposit was revealed in Figure 9 by subjecting the specimen to a 2-second electrolytic etch in 10 per cent oxalic acid at 3 volts and 80 F.

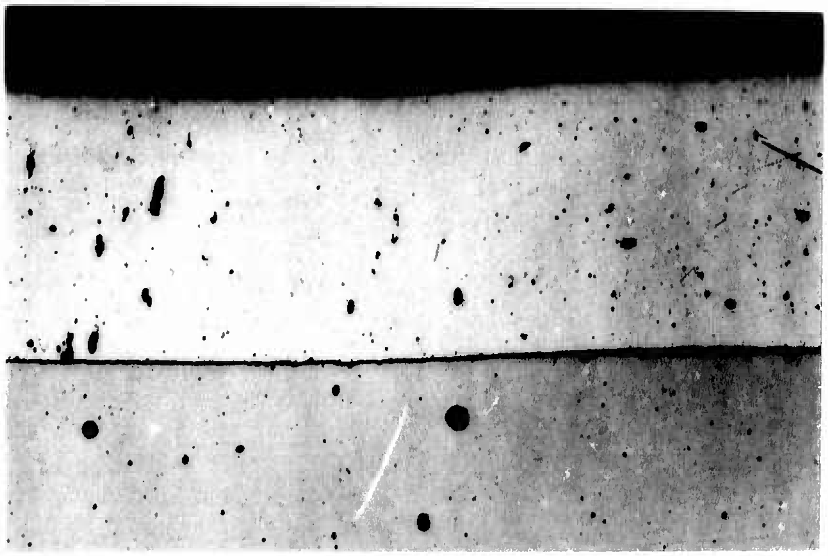
Figure 10 shows a cross-sectional view of the distribution of plate on part of a land and groove.

These tests showed that the 100-gallon bath made up with commercial chemicals was operating as well as any of the smaller volume baths used in earlier work.

Plating of Erosion-Gage Weapon Insert 68X

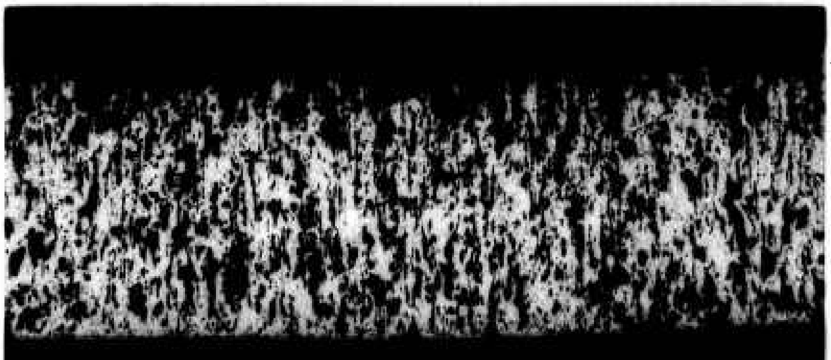
Previous application of the chromium-iron alloy electrodeposit to the SAE 4140 erosion-gage weapon inserts had not shown satisfactory adhesion during firing tests. Insert 68X was plated with a chromium-strike plate. Then chromium-iron alloy plate was electrodeposited over the chromium-strike plate. The plated insert was given a bonding heat treatment at 750 F for 4 hours. This combination had given the best adherence of the plates to 4140 gun-tube steel in previous tests. The sequence of operations is given in Appendix IV.

The insert was greased with a highly refined petroleum sulfonate after initial cleaning. At the time, it was not known that this product, "Petronate", is very difficult to remove prior to plating.



250X As polished 85020

FIGURE 6. CROSS-SECTIONAL VIEW OF CHROMIUM-IRON DEPOSIT BEFORE BONDING HEAT TREATMENT. BASIS METAL IS 4140 GUN-TUBE STEEL



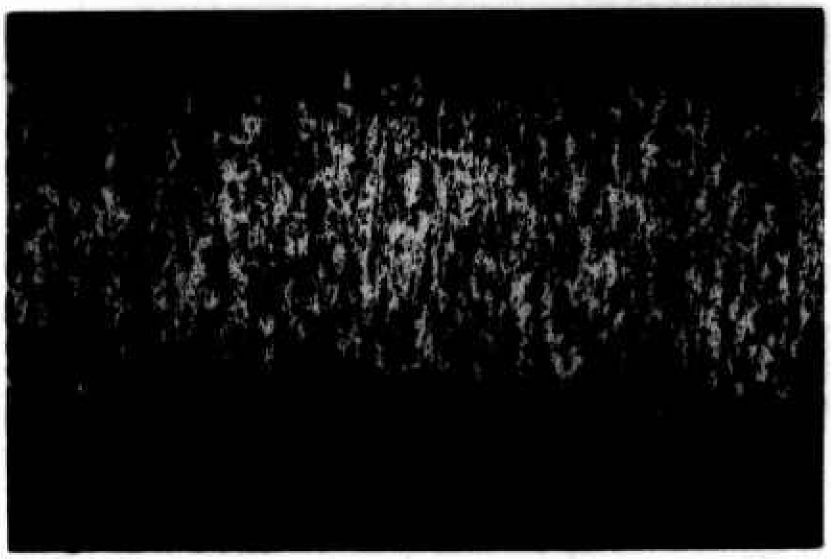
250X Two-second electrolytic etch in 10 % oxalic acid at 3 volts and 80 F 85022

FIGURE 7. CROSS SECTION OF A CHROMIUM-IRON DEPOSIT BEFORE BONDING HEAT TREATMENT

(Note thin chromium-strike deposit at surface of the SAE 4140 basis metal)

250X As polished 85019

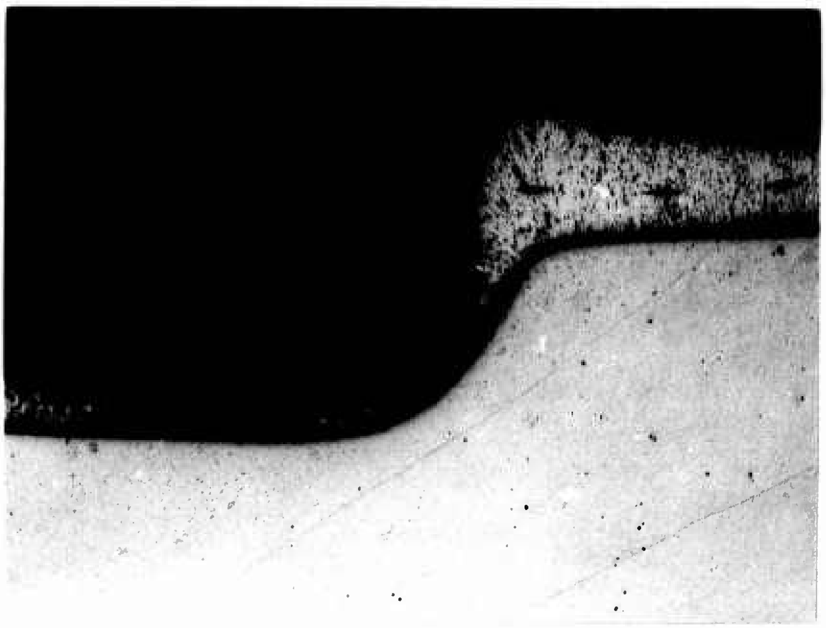
FIGURE 8. CROSS-SECTIONAL VIEW OF CHROMIUM-IRON DEPOSIT AFTER BONDING HEAT TREATMENT OF 750 F FOR FOUR HOURS



250X Two-second electrolytic etch in 10 % oxalic acid at 3 volts and 80 F 85021

FIGURE 9. CROSS SECTION OF A CHROMIUM-IRON DEPOSIT AFTER BONDING HEAT TREATMENT AT 750 F FOR FOUR HOURS

(Note the thin chromium-strike deposit on the 4140 steel surface; also the absence of any change in structure because of the heat treatment)



50X Two-second electrolytic etch in 10 % oxalic acid at 3 volts and 80 F 88544

FIGURE 10. CROSS-SECTIONAL VIEW SHOWING DISTRIBUTION OF THE CHROMIUM-IRON DEPOSIT ON PART OF A LAND AND GROOVE OF THE 40-MM RIFLED BORE SURFACE

(Diamond-shaped indentations are from the Knoop hardness measurements)

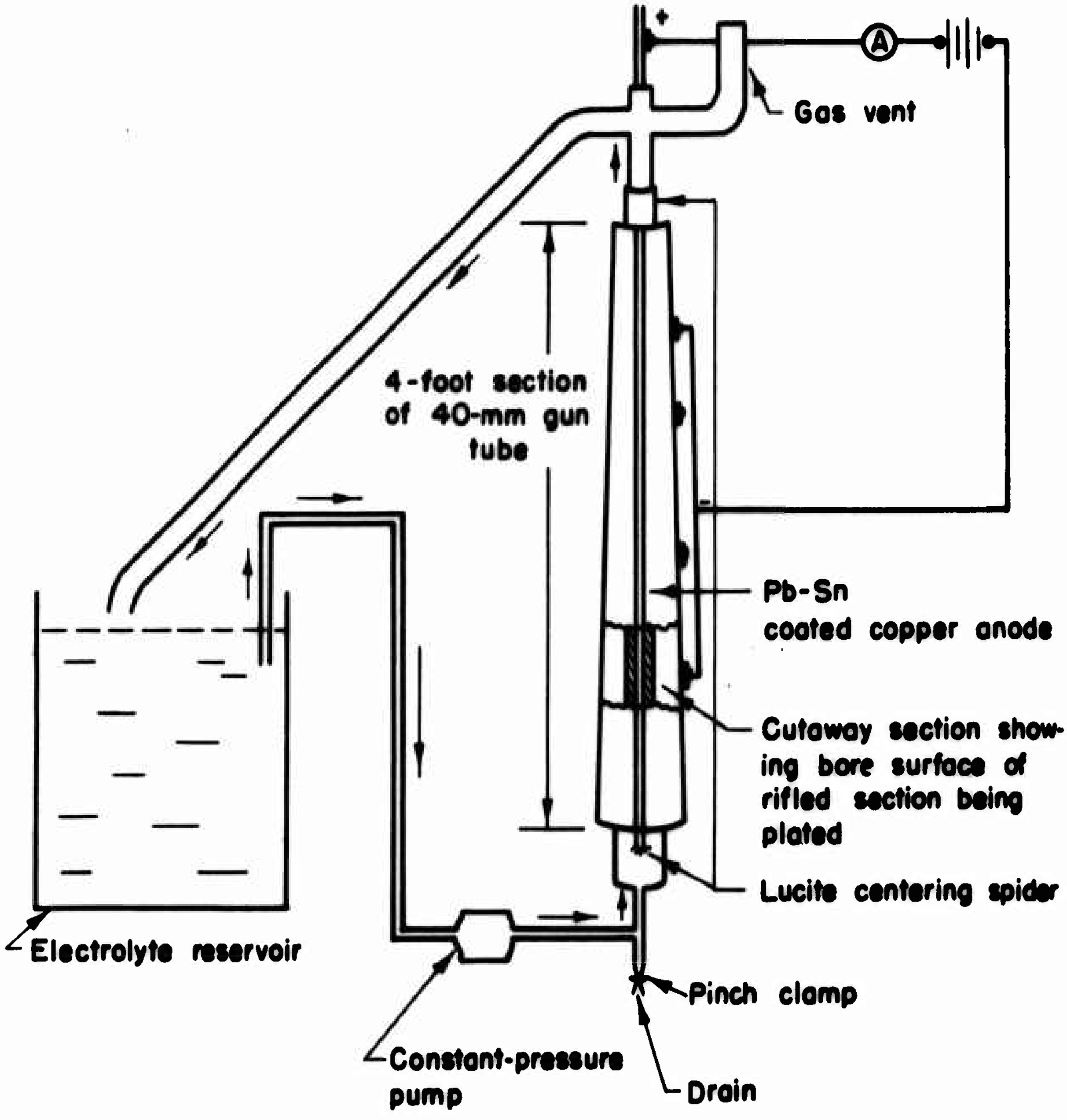


FIGURE 11. SCHEMATIC DRAWING OF PILOT-SCALE PLATING UNIT

A-2089

APPENDIX I

APPENDIX I

Plating of Erosion-Gage Weapon Inserts 61X, 62X, and 63X

Sequence of Operations

1) Pretreatment
   a) Soak clean, 10 g/l alkaline cleaner at 180 - 190 F for 1/2 hour.
   b) Cold-water rinse.
   c) Pumice scrub.
   d) Cold-water rinse.
   e) Activation dip — HCl — 50% by volume.
   f) Cold-water rinse.
   g) Mount tube in fixture and position anode.

2) Nickel-Strike Plating

Bath composition — 250 g/l $NiSO_4 \cdot 6H_2O$
50 g/l H_2SO_4.
Bath temperature — 106 F, cathode CD - 100 amp/sq ft.
Bath treated with Nu Char activated carbon and filtered.
A new portion of the bath was used for each test.

Procedure
   a) Immerse fixture and tube in bath with current on (16 amperes). Strike plate for 5 minutes at 100 amp/sq ft (3 volts).
   b) Remove from bath, rinse in cold water.
   c) Immerse in HCl solution (50% by volume) and mildly agitate for 15 seconds to keep nickel-strike surface activated.
   d) Rinse thoroughly in cold water and place immediately in Cr-Fe bath.

3) Chromium-Iron Plating

Bath composition — same as for Bath No. 5977-39 (Table 2). Bath pH-1.15 measured at 140 F. Bath had been electrolyzed, so all the iron was ferric. Bath also contained a slight trace of hexavalent chromium. The Cr^{+6} buildup from each test was reduced with hydrogen peroxide.

APPENDIX II

APPENDIX II

Formulation and Preparation of the 100-Gallon Chromium-Iron Alloy-Plating Bath

Chemicals Used

MSO (Mutual Chemical Company) SO_2 - reduced sodium bichromate - 24% CrO_3	3.78	lb/gal
Ammonium Sulfate $(NH_4)_2SO_4$	0.42	lb/gal
Magnesium Sulfate $MgSO_4 \cdot 7H_2O$	0.167	lb/gal
Ferrous Ammonium Sulfate $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$	0.0418	lb/gal
Sodium Sulfite $Na_2SO_3 \cdot 7H_2O$	3.78	g/gal
Sulfuric Acid H_2SO_4 (60%)	0.425	lb/gal

Bath Preparation

A glass-lined, 100-gallon plating tank was filled two-thirds full of tap water and heated to 165 F. This was accomplished by passing steam through Duriron heat exchangers suspended in the bath.

The MSO (chromium ammonium sulfate) was added and stirred in. When it was completely dissolved, the required amounts of $(NH_4)_2SO_4$, $MgSO_4 \cdot 7H_2O$, $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$, and $Na_2SO_3 \cdot 7H_2O$ were added. The H_2SO_4 was added as explained in the following section on bath aging.

Bath Aging

A chromium-iron bath made up according to the above formulation has a pH of 2.4 to 2.6 as measured at room temperature. On standing at bath-operating temperature (140F), the various trivalent-chromium

APPENDIX III

APPENDIX III

Sequence of Operations for Plating 3-Inch-Long, 40-Mm Rifled Sections

1. Strip plate in hydrochloric acid (50 per cent by volume of 1.18 sp gr acid) (1:1), rinse, and dry if a plated sample is reused.

2. Heat treat at 400 F for 4 hours.

3. Vapor blast.

4. Soak clean in a 15-oz/gal solution of Anodex for 15 minutes at 180 F.

5. Cold-water rinse.

6. Activation etch in hydrochloric acid (10 per cent by volume of 1.18 sp gr acid) (1:3).

7. Cold-water rinse.

8. Reverse etch in conventional chromic acid plating bath at 100 amp/sq ft for 1 minute.

9. Chromium-strike plate at 240 amp/sq ft for 10 minutes.

10. Cold-water rinse.

11. Immerse in chromium-iron alloy-plating bath with current off for 5 minutes.

12. Apply current at 240 amp/sq ft, 6 volts, and plate for 2 hours, bath temperature 140 F.

13. Rinse in cold water and dry.

14. Heat treat in air at 750 F for 3 hours. Furnace cool.

APPENDIX IV

APPENDIX IV

Sequence of Operations for Plating Erosion-Gage Weapon Insert 68X

1. Soak clean in 15-oz/gal solution of Anodex at 180 F for 30 minutes.

2. Cold-water rinse.

3. Acid dip in hydrochloric acid (50 per cent by volume of 1.18 sp gr acid).

4. Cold-water rinse.

5. Reverse etch in a conventional chromic acid alloy-plating bath at 100 amp/sq ft for 2 minutes at 115 F.

6. Chromium-strike plate at 230 amp/sq ft for 10 minutes at 115 F - 53 oz/gal CrO_3, 0.53 oz/gal sulfate.

7. Cold-water rinse.

8. Immerse in chromium-iron for 5 minutes with current off.

9. Chromium-iron plate for 1 hour at 230 amp/sq ft, bath temperature 140 F.

10. Cold-water rinse and dry.

11. Bonding heat treat in air at 750 F for 4 hours. Furnace cool.

Plating-Fixture Design

Machined Lucite fixtures were slipped over both ends of the insert after the soak-clean operation. A lead-tin-coated, 3/16-inch copper-rod anode was inserted through a hole in the bottom fixture, and pushed up through the insert and out through a hole in the top fixture. During plating, the assembly simply was immersed in the bath.

APPENDIX V

APPENDIX V

Sequence of Operations Used in Plating the Bore Surfaces of 4-Foot-Long, 40-Mm Rifled Gun Tubes

Pretreatment

The pretreatment sequence was complicated by the fact that a thin rust film formed on the bore surface of the gun tubes after vapor blasting. For some reason, the rust inhibitor used in the vapor-blast mixture did not function as it had in previous tests on 40-mm rifled sections.

1. Soak clean in Anodex, 15 oz/gal, at 180 F for 30 minutes.

2. Vapor blast - a cross-sectional view of the nozzle extension used is shown in Figure 11. 600-mesh "Novaculite" was used. Each tube was vapor blasted for approximately 1 hour.

3. Dry without removing vapor-blast sludge. Note: Bore surfaces started rusting within 24 hours even though rust inhibitor had been added to the vapor-blast mixture.

4. Rinse and dry thoroughly.

5. Swab bore surface with "Petronate". Note: Plating of tubes held up several days awaiting the arrival of special plastic tubing for the apparatus.

6. Soak clean, new Anodex cleaner used, 15 oz/gal at 180 F for 30 minutes.

7. Rinse and pumice scrub.

8. Rinse.

9. Hydrochloric acid etch (50 per cent by volume of 1.18 sp gr acid).

10. Rinse.

TABLE 3. EFFECTS OF PRETREATMENT AND HEAT TREATMENT ON ADHESION OF Cr-Fe ALLOY TO SAE 4130 STEEL

			Pretreatment				Cr-Fe Anodic Treatment		Cr-Fe Plating Conditions							
			CrO_3 Reverse		CrO_3 Strike										Adhesion	
Test Number	Cathode Material	Anode Material	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	pH	Temp, F	In With Current On or Off	Description of Deposit	Heat Treat 750 F 2½ Hr	Bend Test	Chisel Test
Bath No. 6526-20 Composition: 453 g/l MSO-2 20 g/l $MgSO_4 \cdot 7H_2O$ 13.5 g/l $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$ 0.5 g/l $Na_2SO_3 \cdot 7H_2O$ 50 g/l $(NH_4)_2SO_4$ 5.5-liter volume																	
6526-28A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	144	—	—	40	240	1.25	140	Off	Very good	Yes	Very good	Very good
6526-28B	SAE 4130 steel	94% Cr, 6% Fe	1.5	96	2	144	—	—	40	240	1.25	140	Off	Very good	No	Very good	Very good
6526-29A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	10	240	0.5	240	40	240	1.21	140	—	Flaked	—	—	—
6526-29B	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	10	240	0.5	144	40	240	1.21	140	—	Very good	Yes	Very good	Very good
6526-29C	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	10	240	0.5	144	40	240	1.21	140	—	Very good	No	Very good	Very good
6526-30A	SAE 4130 steel	94% Cr, 6% Fe	—	—	—	—	—	—	40	240	—	140	Off	Flaked	—	—	—
6526-30B	SAE 4130 steel	94% Cr, 6% Fe	—	—	—	—	—	—	40	240	—	140	Off	Flaked	—	—	—

TABLE 4. (Continued)

Test Number	Cathode Material	Anode Material	Pretreatment: NaOH Reverse Etch: Cathode Current Density amp/sq ft	Time, min	Temp, F	CrO$_3$ Reverse: Cathode Current Density, amp/sq ft	Time, min	CrO$_3$ Strike: Cathode Current Density, amp/sq ft	Time, min	Hardness of Steel: BHN	Rc	Cr-Fe Reverse: Cathode Current Density, amp/sq ft	Time, min	Cr-Fe Plating: Cathode Current Density, amp/sq ft	Time, min	Temp, F	pH	Hydrogen Anneal: Time, hr	Temp, F	Remarks
Bath No 6526-14A Composition: 453 g/l MSO-2, 50 g/l $(NH_4)_2SO_4$, 5.5-liter volume																				
6526-18A	SAE 4140 steel	99% Pb, 1% Ag	–	–	–	–	–	–	–	–	13	–	–	300	30	140	1.3	–	–	Deposit appeared good but flaked when chisled.
6526-19A	Ditto	Ditto	–	–	–	96	0.5	144	5	–	13	300	0.5	–	–	–	–	–	–	1.1 g/l Na_2SO_3 added to bath. A black smut appeared on cathode after reverse in Cr-Fe.
6526-19B	"	"	–	–	–	96	0.5	144	10	–	13	300	0.5	–	–	–	–	–	–	Chromium strike withstood reverse etch in Cr-Fe bath with no appearance of black smut.
Bath No. 6526-20 Composition: 453 g/l MSO-2, 50 g/l $(NH_4)_2SO_4$, 20 g/l $MgSO_4 \cdot 7H_2O$, 13.5 g/l $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$, 1.0 g/l $Na_2SO_3 \cdot 7H_2O$																				
6526-20A	SAE 4140 steel	Pb-Sn-coated Cu	–	–	–	200	0.5	300	10	–	27	300	0.5	300	30	140	–	–	–	Excellent adhesion after bonding heat treatment at 750 F for 2-1/4 hours. Deposit flaked before heat treatment when chiseled.
6526-21A	Ditto	Pb-Sn-coated Cu	–	–	–	200	0.5	300	10	–	13	300	0.5	300	30	140	1.45	–	–	Deposit had only fair adhesion.
6526-22A			–	–	–	–	–	–	–	–	13	–	–	410	30	140	1.5	–	–	Deposit blistered slightly.
6526-22B	"	Pb-Sn-coated Cu	–	–	–	200	0.5	300	10	–	13	240	0.5	410	30	140	–	–	–	Deposit flaked when chiseled before heat treatment at 750 F for 2-1/4 hours but withstood chisel test afterwards.
6526-23A	"	Pb-Sn-coated Cu	–	–	–	–	–	–	–	–	13	–	–	375	30	140	–	–	–	Deposit flaked before and after heat treatment.
6526-23B	"	95% Cr, 5% Fe	–	–	–	–	–	–	–	–	13	–	–	410	30	140	1.8	–	–	Deposit blistered.

TABLE 5. EFFECT OF VARYING THE HEXAVALENT-CHROMIUM CONTENT ON THE CATHODE CURRENT EFFICIENCY

			Pretreatment												
			CrO_3 Reverse		CrO_3 Strike		Cr-Fe Reverse		Cr-Fe Plating Conditions				Amount of Cr^{+6}, g/l		
Test Number	Cathode Material	Anode Material	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	Time, min	Cathode Current Density, amp/sq ft	pH	Temp, F	Before	After	Cathode Current Efficiency*
Bath No. 6526-20 Composition: 453 g/l MSO-2 13.5 g/l $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$ 50 g/l $(NH_4)_2SO_4$ 20 g/l $MgSO_4 \cdot 7H_2O$ 0.5 g/l $Na_2SO_3 \cdot 7H_2O$ 5.5-liter volume															
6526-32A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	1.31	140	–	.169	47.2%
6526-34A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	1.35	140	.557	.66	46.6%
6526-35A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	–	140	1.06	1.21	45.4%
6526-36A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	–	140	1.54	1.63	44.8%
6526-36B	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	–	140	1.95	2.12	42.8%
6526-37A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	–	–	–	140	2.49	2.75	42%
6526-37B	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	1.28	140	2.84	2.99	41.8%
6526-38A	SAE 4130 steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	–	140	3.28	3.38	40.6%
6526-39A	Pb-Sn-coated steel	94% Cr, 6% Fe	0.5	96	2	240	–	–	40	240	1.30	140	3.38	–	40.6%

* All efficiencies were based on the pure chromium deposition in g per amp-hr, which is 0.646 g/amp-hr for trivalent chromium.